STATE ENGINEERING EXPERIMENT STATION

The Research Engineer

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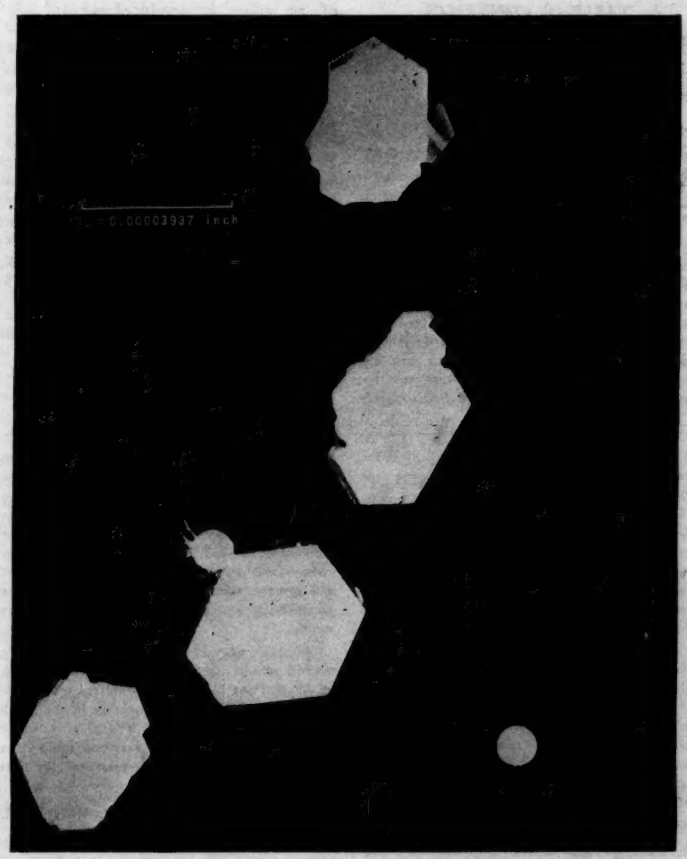

The Research Engineer

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TABLE OF CONTENTS

Electron Microscopy 3

Life and Death at Low Temperatures . . 5

Exposure Testing of Exterior Paints . . . 7

Recent Station Publications 9

Index to 1950-1951 Issues 19

Cover: Electron Micrograph of Kaolin

QUANTITATIVE RESEARCH

The discovery of a new scientific phenomenon is usually the result of qualitative research—the attempt to find out *what* happens under certain conditions. Before the phenomenon can be put to practical use it is necessary to know much more about it—*how much* of an effect is produced under just which *precisely measured* conditions—this being the province of quantitative research.

As Prof. Kethley points out in his article beginning on page five, qualitative investigations leading to a new discovery can be, and often are, performed by an individual experimenter working with inexpensive equipment. But to provide the knowledge essential for control and application of a phenomenon requires systematic, quantitative study of its fundamental governing laws. This is often a costly procedure involving teams of scientists and elaborate equipment. Yet only when these laws or limitations are known can practical potentialities be assessed.

Continued on Page 24

The growing trend of industry to move South promises an economic renaissance for Georgia and her neighboring states. World War II experiences demonstrated the capability of Southern labor to staff plants requiring skills long considered a unique resource of established industrial regions. Thus fell the last barrier to development of the South's extensive natural resources.

As industries migrate to our area and as native industries develop to process our abundant raw materials, both mineral and agricultural, the demand for technically trained manpower will increase markedly. Men with special knowledge will be needed at all levels—skilled labor, technicians, engineers, research scientists and executives.

Georgia Tech is well prepared to supply men of the type required. Last June, 1,541 bachelor and 108 master degrees were awarded by Tech's full-time engineering and science schools. Enrollment for evening school college credit, refresher, certificate and adult education courses given by our Engineering Extension Division totaled 2,228 for the 1949-50 session. This Division also provides technical training for employees in their own plants, conducts short courses and conferences on industrial problems and operates the Southern Technical Institute, a most fertile source of the numerous technicians needed.

Although over 70 per cent of Georgia Tech's alumni now reside in the southeast (8,961 out of 19,272 live in Georgia), increased industrialization will encourage the retention in the South of more Tech graduates, young men whose education, perseverance and ambition equip them for roles in our economic progress and for lives of constructive citizenship.

BLAKE R. VAN LEER,

President, Georgia Institute of Technology

ELECTRON MICROSCOPY

By L. A. WOODWARD*

The use of the electron microscope in the study of very small objects—those beyond the range of the optical microscope—has received considerable publicity during recent years, although the principles involved and the techniques used in the preparation of samples are not at all well known. An over-all picture of the operation of the electron microscope and a discussion of some of its limitations are presented in this article, which will be followed (in a later issue) by an article describing the research uses of the Georgia Tech instrument.

The invention of the optical magnifier in the 17th Century opened new vistas for scientific investigators of that day. Following this, improvements in lens systems reduced further and further the limiting size of the smallest object that could be clearly viewed. However, after a time, continued developments failed to decrease that minimum size which was finally reached, and the wave theory developed during the latter part of the 19th Century finally indicated that the difficulty lay not in the lens systems but in the illumination system, namely, visible light.

Because of the relatively long wave length of the electromagnetic radiation that composes the visible spectrum, the best optical systems are not capable of forming a clear image of an object smaller than 0.15 micron (0.15×10^{-6} meter, or about six millionths of an inch). The use of invisible ultraviolet light improves this resolution, but only by a factor of about two. Using X-ray radiations, which possess even shorter wave lengths, would improve this resolution still further, but a lens system that will form an image with these has yet to be devised. However, because of their short wave length and great penetrability, X-rays are used in diffraction, which employs the reflection of the rays off individual atoms in crystal lattice structures. This indirect method gives considerable information about very small distances and particle sizes.

The same wave theory of light that pointed out the limits of visible light systems also indicated that a stream of rapidly moving electrons, hitherto considered as simply a procession of discrete particles, should exhibit a wave-length characteristic depending on the speed with which the electrons were moving. Experimental investigations following the approach of X-ray diffraction techniques verified this aspect of the theory and led to the acceptance of associating wave lengths with these so-called matter waves. The wave lengths readily attainable for electron velocities are many thousands of times smaller than the smallest wave length in the visible light range. Since a moving charged particle such as an electron constitutes an electric current, previous studies of forces on current-carrying wires in magnetic fields led the way to the development of "lenses" which could bend the electron beam just as the lens in a reading glass bends a light beam.

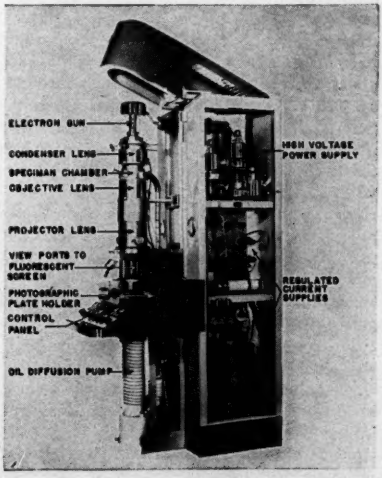

Figure 1. The Georgia Tech Electron Microscope, with covers removed.

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Thus, a microscope using an electron illumination system and properly shaped magnets as lenses came into being, pushing back by a factor of over 20 the limiting size of the smallest object that could be clearly viewed. This improvement, however, was not gained without considerably increasing the complexity of the procedure necessary to investigate the previously unexplored world which lies beyond the reach of the conventional microscope. It is the purpose of this paper to discuss the capabilities of the instrument developed and some of the limitations that are necessarily inherent in it; a later paper will demonstrate some of the uses of the electron microscope installation here at Georgia Tech.

DESCRIPTION AND OPERATION

Figure 1 is an "opened-up" picture of the R.C.A. Model EMU-2 Electron Microscope in the Georgia Tech Engineering Experiment Station. The microscope proper consists of a metal tube about three feet long and several inches in diameter. This tube is made in sections (some of which are the magnetic lenses), fitted together with numerous vacuum seals in order that the column (tube) can be evacuated to a low pressure. At the top of the column is an electron gun, which consists of a heated tungsten filament that produces electrons, and plates that accelerate the electrons through 50,000 volts and direct them into a narrow beam. This is the only linear acceleration that the electrons receive, and, in order that they will continue on down the column without too many collisions with gas molecules, it is necessary to reduce the number of molecules present. A vacuum of around 0.1 micron of mercury pressure, about one ten-millionth of normal atmospheric pressure, is sufficient to reduce these collisions to a tolerable number.

The electron beam now passes through the condenser lens that controls the illumination of the specimen. This lens consists of an iron-clad solenoid with a small aperture in the center. An electrical current passing through the coil controls the strength of the magnetic field produced. Varying this current changes the effective focal length of the lens, achieving the effect of controlling the intensity of the electron beam that reaches the specimen.

The beam now passes *through* the specimen. It is important that this fact be remembered, because of the requirements that it puts on the types and conditions of the samples to be studied. The passage of the electron beam through the specimen produces a "shadowgraph" of it. Where a portion of the sample either absorbs or scatters the electrons, the part of the beam passing through such a portion has fewer electrons than the part which passes through an area of the sample having less effect on the electrons. Specifications for good samples are discussed later in this article.

The objective lens of the electron microscope now forms an enlarged image of the "shadowgraph." In construction, this lens is similar to the condenser lens and, as before, focusing is accomplished by varying the current through the electrical coil. The image produced is in turn magnified by a similar lens called a projector lens, and a second image is formed on a fluorescent screen below this lens. Thus, the enlargement of the "shadowgraph" is produced, as in a compound light microscope, by the multiplication of the magnification of the objective lens and the projector lens.

Immediately below the fluorescent viewing screen is a photographic plate. After the operator has selected the particular part of the sample which he wishes to record and has adjusted the focus, the screen can be lifted and the same electrons that were activating the fluorescent material can expose the photographic plate.

The auxiliary equipment to the microscope proper consists of vacuum pumps to maintain the necessary column pressure, a high voltage supply for electron acceleration, accurately regulated current supplies for the various lenses, and a control panel.

By changes in the assemblies of the objective and projector lenses, and by variation of the current in the projector lens, a range of over-all magnification from 75X to 22,000X may be achieved. There is rarely any reason for using the lower ranges of magnification, as this region is adequately covered

Continued on Page 10

LIFE AND DEATH AT LOW TEMPERATURES

By T. W. KETHLEY*

In recognition of their research which led to the publication of several papers on aspects of the preservation of foods by freezing, T. W. Kethley, W. B. Cown, and F. Bellinger, last spring, received the 1950 First Faculty Award of the Georgia Tech Sigma Xi Club. In addition to receiving a monetary reward, the winner each year is privileged to address the faculty and friends of Georgia Tech, on a subject of his own choice, at a Sigma Xi lecture held during commencement week ceremonies. Professor Kethley chose as his subject "Life and Death at Low Temperatures," a phase of his current research and a topic which, fascinating in itself, also permitted pertinent discussion of the philosophy of research.

The study of low temperatures has a fascination for many, in addition, often, to very practical aspects. In nature, low temperatures are encountered in the arctic regions and (in winter) in the temperate zones; by man's artifice, they are also available at command.

*Research Assistant Professor.

At Georgia Tech, our interest in low temperatures first arose from studies on the fundamental factors involved in the preservation of foods by freezing. Incidentally, the development of our work in this latter field might well serve as a case history in the study of the relationship between fundamental and applied research. Our initial objectives related to applied problems,

Figure 1. T. W. Kethley using equipment for the study of the subcooling characteristics of bacterial suspensions.

but it was soon decided that no real progress would be made until we had obtained a more thorough knowledge of the basic problems involved.

However, in planning our work, we found no satisfactory methods for evaluating our studies, and were thus confronted with the problem of devising our own techniques. In order to accomplish this logically, it was felt that a knowledge of the processes involved was essential. We therefore laid out a two-pronged, broad program of research involving (1) a study of processes, the results of this study to be applied to the development of objective methods for evaluating changes in frozen foods; and (2) a study of causes, the results of this study to be applied to the design of equipment for the freezing preservation of foods under optimum conditions.

Interest in the subject of the present discussion—life and death at low temperatures—arose from the latter phase of the above program. It appeared that knowledge of the exact nature of the cause of death by cold to cells and tissues might be of value in the determination of the nature and causes of the changes which follow the death of the cells of food tissues.

Evidence thus far obtained, however, indicates that the primary causes of cellular death at low temperatures may well be separable from the causes of further physical changes induced in dead cells by freezing and freezing storage. The evidence to support this contention is rather meager, but the possibility is sufficiently significant to warrant examining all evidence in the light of two sets of causations, one to account for cellular death and another to account for deteriorative changes following death. In the material covered in this paper, however, only the question of the causes of death at low temperatures will be considered.

FREEZING OF HIGHLY ORGANIZED BODIES

Man has for centuries been intrigued by reported observations that certain forms of life can be frozen solid and then revived at a later date following thawing. Tales of this kind have been handed down from past generations, but the first recorded scientific attempt to test such a possibility was that of Reamur, who in 1726 reported that caterpillars could be frozen quite solid and yet would show activity when thawed. Further, in modern times, there are recurring stories of survivors of past ages being preserved intact in ice masses—of the flesh of mammoths, ages old, having been maintained in an edible condition. We even have a supposed scientist, one Kapterev of Russia, reporting that crustaceans which he unearthed from deep in permanently frozen soil had revived when thawed. Kapterev thought that the age of the layers of soil from which these crustaceans were taken should be reckoned in thousands of years. It is perhaps appropriate that these specimens were reportedly dug from the valley of the Great Never River!

The myth of the revived frozen goldfish is a modern one—the story that these fish can be frozen in liquid air until sufficiently brittle to be shattered by a blow of a hammer, yet will be able to swim if thrown into water. All of these tales deal with highly organized forms of life. However, it is extremely doubtful that such life can be preserved by freezing; the breakdown of communications within the highly organized body usually causes somatic death long before the individual cells have ceased their activity.

More recent investigations have furnished us with rational explanations of most of these myths. It has been observed that the forms of life most commonly thought to survive freezing — caterpillars, frogs, and fish—enter a state of rigidity when their body temperatures approach freezing. Most present-day workers feel that earlier observers confused this rigidity with the frozen state. Furthermore, earlier workers did not take into consideration the length of time required to cause the internal temperature of a frog or fish to drop to or below the freezing point; in most instances, the times of exposure to cold were insufficient to insure actual freezing. Contemporary workers have shown that death ensues when the internal temperature of a frog or

Continued on Page 12

EXPOSURE TESTING—A GUIDE FOR IMPROVEMENT OF EXTERIOR PAINTS

By W. R. TOOKE, JR. *

The beauty and durability of exterior house paints have been greatly improved during recent years through the development of new paint-making materials and new formulations of these materials. Paint exposure tests have contributed materially to these advances by providing a basis for evaluation of the new with the old and by indicating the types of failure which occur first under given exposure conditions. The following article describes the Georgia Tech exposure testing program—the methods, equipment and techniques employed.

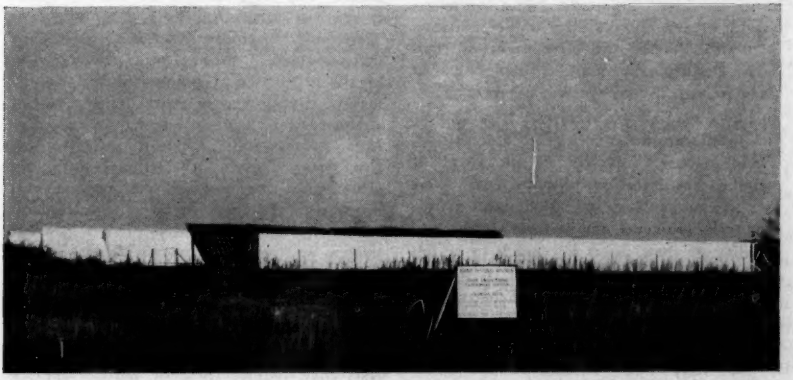
Paint exposure tests are made to obtain data on observable changes in the appearance and properties of paint films subjected to weathering under reasonably well defined and controlled conditions. Series of exposure tests may be designed to compare various paints or to study the effects of variations in composition, manufacture, application and exposure conditions. In general, the test procedure includes collection or preparation of test paints, application of these paints to selected panels or test structures, exposure of the painted panels to weathering and recording of the weathering effects at periodic intervals. The data obtained from properly conducted exposure studies contribute greatly to the development of materials, formulations and practices that will produce paints of improved durability and beauty.

House Paint Progress

The paint industry has made considerable progress in the improvement of exterior house paints during the last two decades. Van Loo[1] has illustrated the comparative magnitude of these improvements with a series of photographs of painted panels having four years' exposure. A formulation typical of those manufactured in 1927 indicated virtually complete film failure, while a postwar formulation was still in excellent condition at the end of the four-year period.

Exposure studies have contributed materially to developments in paint technology. The two-coat (special primer-top coat) system of paint application, the use of mixed pigment paints, and paint formula-

* Research Assistant.



About 3,000 square feet of test panels can be exposed at Tech's Paint Testing Station located just off the Marietta Highway ten miles north of the campus.

tors' increased awareness of the individual contributions of the various pigment and vehicle materials to the properties of the paint film are examples of these developments.

The quality and uniformity of pigment materials have been considerably improved, and new types of pigments have been developed. Among high hiding-power pigments, titanium dioxide has achieved a dominant position. The single-pigment-in-oil type paint is now almost obsolete, and realization that judicious use of extender pigments actually promotes film durability has gained a new respectability for these materials.

The technology of vehicle materials (oils, resins, driers and thinners) has also advanced. Many new synthetic and modified oils and resins have been developed, but these materials have been used in house paints only in relatively small quantities. Favorable results have been obtained with some of the synthetics, however, and more widespread use of these materials may be predicted.

The shortage of linseed oil during World War II forced manufacturers to use a "restricted-oil" type vehicle in which the actual oil content was cut by one-third or more. The composition consisted of approximately equal parts of raw oil, bodied oil and thinner. Paint technologists were somewhat surprised to discover that these "restricted-oil" paints proved superior to prewar paints in many cases. However, being conservative by nature, the majority of paint manufacturers increased the oil content of their paints slightly as a "margin of safety" after the wartime oil shortage was over.

The action of paint driers in catalyzing film solidification has been given much study. These materials, usually in the form of metallic lineoleate or resinate salts, were formerly prepared by individual paint manufacturers.- However, the variable activity and stability of these driers left much to be desired. Accordingly, research led to the development of new and improved driers based primarily on naphthenates and octoates. The manufacture of these driers has become a specialized industry, and most paint manufacturers buy prepared driers rather than making them in their own plants.

Volatile thinners for house paints have attracted comparatively little research attention. For economic reasons, turpentine, the traditional paint thinner, has been virtually replaced by mineral spirits. These two materials seem to perform almost identically in exterior house paints; however, little actual data have been published on this subject. Thinners have been used in much greater quantities in the manufacture of exterior house paints since the adoption of the "restricted-oil" type vehicles previously mentioned.

Thus from the accelerated progress of recent years new and improved paints have evolved. A typical present-day exterior house paint formulation may be described in general terms as a mixed pigment paint with slightly less total oil than prewar paints, but with a higher percentage of heavy bodied oil and thinner. It may or may not contain a small percentage of natural or synthetic resin. This paint is usually packaged at the proper application consistency, and it therefore requires little or no reduction or thinning on the job.

Despite the substantial paint progress already achieved, research will continue to produce improvements. The great complexity of paint mixtures has, for the most part, necessitated empirical approaches to the solution of immediate problems. Comparatively little knowledge of the fundamental chemistry and physics of film formation, adhesion and degradation has been gained. It may be noted, however, that in recent years more attention has been directed to these problems. For example, investigation of the properties of pigmented and unpigmented films prepared from highly purified compounds is being conducted by the *National Federation of Paint and Varnish Production Clubs*. Other organizations are also conducting research on fundamental problems. As these investigations lead to more generalizations and improved theories, *science* will more completely replace *art* in paint-making.

EXPOSURE TESTING TECHNIQUES

Many conditions and variables markedly affect exposure test results. Since individual tests may require three or more years for

Continued on Page 16

RECENT STATION PUBLICATIONS

The following publications have been released by the Station since those reported in the March, 1950, issue of THE RESEARCH ENGINEER.

REPRINTS

R. S. Ingols, *A Study of B.O.D. With Time as the Measurable Variable*. Georgia Institute of Technology, Engineering Experiment Station Reprint No. 39, 1950. 6 pages. Twenty-five cents.

This article, reprinted from the November, 1949, *Sewage Works Journal*, reports the results of an investigation on the possibility of developing a short-time B.O.D. test in which time would be the measurable variable. This was not successfully accomplished, however, apparently because of the excessive time consumed (lag period) during which the bacteria make the necessary changes in the redox potential of the sample and because of the low initial redox potential of some of the samples, which resulted in the immediate decolorization of the indicator. The experimental apparatus and procedure are described and the conclusions are presented primarily as a study of the manner in which the redox potential of sewage samples obtained from several sources changed during incubation with either free or combined oxygen. *A.C.R.*

T. W. Kethley, W. B. Cown, and F. Bellinger, *Freezing Time-Leakage Relationship for Strawberries;* and *An Estimate of Thermal Conductivities of Fruits and Vegetables*, Georgia Institute of Technology, Engineering Experiment Station Reprint N. 40, 1950. 5 pages. Twenty-five cents.

The relationship between leakage from strawberries and the time required for their freezing has now been determined, according to the first of these articles, reprinted from the January, 1950, issue of *Food Industries*. Berries which are cooled to 0° F. in 15 minutes or less will be of essentially the same quality, but berries frozen less rapidly will show a decrease in quality as the time of freezing increases. In the second paper, reprinted from the January, 1950, issue of *Refrigerating Engineering*, procedure and results are presented for successfully estimating the time-temperature data for cooling and freezing experiments, a prime necessity in studying certain biological and physical characteristics which are affected in the freezing of foods. *A.C.R.*

R. S. Ingols, J. C. Hildebrand, and G. M. Ridenour, *Measuring the Strength of Sewages and Trade-Wastes—B.O.D. or O.C.?* Georgia Institute of Technology, Engineering Experiment Station Reprint No. 41, 1950. 7 pages. Twenty-five cents.

This paper, reprinted from the January, 1950, *Water and Sewage Works*, presents in detail a discussion of the advantages and disadvantages of both the O.C. and B.O.D. techniques for measuring the strength of sewages. The authors conclude that much more can be learned about the sewage or industrial waste sample from a careful comparison of the O.C. and B.O.D. values than can be learned from the use of either test alone. *A.C.R.*

CIRCULAR

E. P. Cofield, Jr., *The Solvent Extraction of Oilseed. An Informational Survey*, Georgia Institute of Technology, Engineering Experiment Station Circular No. 28, 1950. 103 pages. One dollar and fifty cents.

This survey, concerned primarily with the process of solvent extraction of oilseed—its capabilities and limitations, the types of equipment used and under development, the applicability of the process to various Georgia-grown oilseed (and others), its economic aspects, and the research opportunities that it offers—was prepared to answer the need for a comprehensive coverage of the technical and economic aspects of this rapidly developing field. In addition, various oil-bearing materials that are of economic importance are discussed, as are the locales in which they are grown and processed. The chemistry of the compounds commonly found in such materials and the structure of typical seed and fruits are also reviewed. Other commercial methods of processing oleaginous materials are described briefly, and a survey is included on the existing and potential commercial uses of oils and other products derived in their processing.

ELECTRON MICROSCOPY

Continued from Page 4

by light microscopes; alternately, magnifications of over 15,000X impose serious limitations on the types of samples used. The range from about 4,000X to 13,000X is most often used and can be changed through ten steps by the turning of a single knob. Micrographs on the photographic plates can in turn be enlarged, and prints up to 9X made, subject to the usual photographic limitations of film grain size and clearness of focus on the plate. Thus, print magnifications of 100,000X, and beyond in special cases, are obtainable by a combination of electron microscope and photographic enlargement.

SPECIMEN SELECTION AND PREPARATION

The selection and preparation of specimens involves many special problems, so that each particular case has to be handled individually. Sometimes, in fact, seemingly similar substances require decidedly different preparation. Certain general considerations are, however, common to all. As mentioned, the electron microscope column is evacuated, so that the electron beam will not be scattered by collisions with gas molecules. Therefore, the specimen must be of such a type that it can be viewed in this vacuum. It is not possible to construct an air lock to maintain the sample at normal pressures, since no material is known that would be strong enough and still be transparent to the electron beam. This means that most living organisms and tissues would be drastically changed when placed in the column. Materials with high vapor pressures would be unsuitable for the same reason.

Also previously mentioned was the fact that the electrons must pass *through* the sample. This requires that the sample be thin, usually a few tenths of a micron if very large, or, if very small, it must be mounted on a suitable substrate. Although the specimen is, in effect, bombarded by the electrons, it must not change because of this unless such change is involved in the information that is desired. When the beam passes through the specimen, there is considerable local heating, which may change the vapor pressure to a point where the sample evaporates. Many crystalline materials are affected by such bombardment.

If the sample is a fine powder and information such as particle shape and size distribution is desired, some means must be found to disperse the specimen in or on the substrate. This is often by no means an easy problem when the particles are very small, tenths of a micron or under in size, and sometimes it is impossible to do an entirely satisfactory job. If the range of particle sizes is large, it is preferable to find some means to divide the sample into smaller size-range fractions, otherwise dispersion is more difficult and details on the electron micrograph may be obscured. Stoke's Law settling affords a means of effecting such division, as does the centrifuge.

Samples should be nearly chemically neutral, otherwise difficulty is encountered in the preparation of the substrate.

The principal means of attaining contrast in the image formed is the selective scattering of the various regions of the specimen. This scattering is a function of the atomic number of the atoms involved, and for this reason materials containing the lighter atoms do not produce as good a picture as those with heavier atoms.

In summary, a specimen must be able to stand high vacuum and electron bombardment, be thin enough for the electron beam to pass through it or small enough so that the narrow beam necessary for high magnification may pass around it, and, unless prepared as indicated below, have a high enough atomic number to give sufficient contrast.

A shadowgraph is necessarily two dimensional. Conventional photomicrographs do not, therefore, directly indicate the complete shape of the particle. Certain specimens in which most of the particles are similar may aggregate in such a way that some of the pieces are caught together and have different sides exposed to the beam. Then, evaluation of a series of plates may give sufficient data to establish the complete particle shape. Samples of crystalline materials which have considerable homogeneity of size lend themselves to this approach.

Also, at times it is possible to take one

exposure of a specimen and then by increasing the beam current cause the substrate to break. If conditions are suitable, the particle, still adhering to the curling substrate, may turn through 90°, affording an opportunity for a second exposure. Although this occurrence is, of course, largely a matter of fortunate chance, since there is no means of controlling the rotation, there are several examples of it cited in the literature, and it has been accomplished at Georgia Tech on several occasions.

REPLICA TECHNIQUES

There are several ingenious methods of specimen preparation which are used to overcome some of the limitations mentioned above. These include replica techniques and heavy metal shadowing. A material such as a metal surface that cannot be cut thin enough to pass the necessary electron beam may be viewed by making a thin transparent replica of its surface that can pass sufficient electrons. Materials that are used for substrates, such as Collodion or Formvar (polyvinyl formal), or evaporated films of silica can be used for this purpose. Usually, the replica requires the metal shadowing treatment described below to attain sufficient contrast. Certain biological samples, when properly fixed, can also be examined by the replica method, thus eliminating exposure of the sample to the vacuum.

SHADOW CASTING

Of considerable usefulness in indicating the third dimension, as well as increasing image contrast and improving definition, is the technique of shadow casting. In this process, a heavy metal, usually gold or chromium, is evaporated in a vacuum and allowed to fall obliquely on a suitably mounted specimen. Because of the lack of gas molecules, the metal propagates in straight lines from a very small source. Any irregularities in the substrate—usually the specimen itself—cause the metal to be shielded from a small region beyond the sample particle away from the source, depending on the angle used and the height of the sample. When electron micrographs are made from a shadowed specimen, the regions shielded from the metal scatter fewer electrons and so appear darker on the negative plate. If a positive plate is then made, and a print made from it, these shielded regions appear as dark shadows. From an accurate knowledge of the shadow angle, usually obtained by introducing a standard particle, measurement of the shadow can yield a definite indication of the height of the particle. In addition to this, any surface irregularities on the particle itself are brought out, and, because the metal deposits around the outline of the particle on at least three sides, definition is improved. Specimens with too low an atomic number for normal study may be viewed by the form of the metal coating they receive.

CALIBRATION

The above mention of a standard particle calls to mind a rather interesting instance of chance production and alert observation. The Dow Chemical Company sent samples of a latex solution to the University of Michigan for particle size distribution analysis on their electron microscope. There, it was found that over 99.99 per cent of one sample was composed of perfect spheres of uniform size. Portions of the remaining sample were sent to electron microscope laboratories all over the country with the request that they determine the size of these spheres. Fourteen independent measurements agreed that the size was 0.2590 ± 0.0025 micron.

These spheres filled a long standing need for a standard particle for calibration in the range that hitherto had presented a difficult problem. The focal lengths of the magnetic lenses depend on the current passing through the electrical windings, and this current is difficult to control in a completely satisfactory manner. In addition, the effects of the pole pieces on the magnetic field change with use. Therefore, the magnification obtained is not fixed, as in a set of light lenses, but changes with time and with use. The changes are not large, but accurate work on the smaller particles requires constant calibration. The usual procedure, formerly, was to make a picture of an easily recognized particle and then measure it on a calibrated light microscope. However, because of the difference in range of the two instruments, this did not give too satisfactory results in

the range where the electron microscope was most valuable. The discovery of the uniform latex spheres provided a calibrated particle in the most desirable size range, one that could usually be introduced along with the specimen and thus calibrate both the micrograph plate and any subsequent enlarged prints.

Incidentally, it is not known why this particular latex sample turned out as it did, and there is no way of controlling the process to produce more. However, enough of the sample is available to serve everyone desiring some, and, since such a tiny amount is used for a given picture, the five-cubic-centimeter bottles distributed (without charge, by the way) should last a rather active laboratory at least 100 years.

During the five years that the Georgia Tech electron microscope has been in operation, it has been used extensively for research, both in the Engineering Experiment Station and for outside organizations. It has proved of particular value in evaluating samples from a project which dealt with the electrolytic preparation of activated manganese dioxide, as well as in studies on a variety of different materials such as clays, pigments, minerals, and gold sols. Without doubt, this electron microscope will continue to be of increasing value to research in many fields as other industrial and institutional groups learn of its function and its availability at Georgia Tech. Some of the studies already performed will be described in a future article.

LIFE AND DEATH AT LOW TEMPERATURES

Continued from Page 6

a fish is lowered enough to induce freezing.

The goldfish myth deserves special consideration. Luyet mentions that he knew a juggler who used the freezing and thawing of goldfish as part of his act, and observes: "animals to be broken before the audience should be left for 35 seconds in liquid air, and those to be withdrawn, less than 10 seconds." Such a slight difference in time of immersion is easily masked by the patter of the professional showman. It is of further interest that Luyet thoroughly investigated the matter and obtained no sign of recovery from fish immersed for more than 15 seconds in liquid air, although he discovered that immersion for at least 35 seconds was required to render the fish breakable.

FREEZING OF MICROORGANISMS

Turning from more highly organized forms of life to the consideration of the effect of cold on single-celled organisms—the so-called microorganisms—one enters a different realm. It was observed and recorded as early as 1879 that the organisms responsible for yellow fever could survive subfreezing temperatures for long periods of time, and in 1885 an epidemic of typhoid fever was traced to cultures of the organism which had remained frozen for weeks. A few years after this, various medical men became impressed by the destructive power of liquid air on plant life, and extravagant claims were made for the germicidal power of liquid air. In order to test these claims, Mazyck P. Ravenal, an M.D. of Philadelphia, carried out certain most ingenious experiments in the year 1899. He obtained a small quantity of liquid air from Professor Barker of the University of Pennsylvania and demonstrated conclusively that some pathogenic microorganisms could survive cooling to a temperature of at least —190° C.

The techniques employed by Ravenal were extremely simple, yet most effective. Taking sterile silk threads, he dipped these into the cultures of his organisms and then quickly immersed the impregnated threads into the liquid air. The threads were left in the liquid air for varying lengths of time, up to three hours. At the end of the selected interval of time, the threads were removed from the liquid air and immediately placed in nutrient solutions in an incubator. To quote the doctor: "In no case were the bacteria killed, nor could any in-

hibition even be detected, all cultures growing as vigorously as the controls."

Although Ravenal's experiments were not chronologically the first to demonstrate the survival of bacteria at extremely low temperatures, they were the first recorded with sufficient completeness of detail to allow us to reconstruct them. Pictet of Geneva had published papers in 1884 and 1893 on the action of extreme cold on microbes, but certain details of his experiments were not recorded. Furthermore, the experiments recorded by Ravenal are complete within themselves—they can, as it were, stand by themselves without the need of external support; controls were included, and all pertinent conditions were specified. In addition, these experiments recommended themselves because of their extreme simplicity, both as to equipment and technique. Here was a model experiment, an ideal case of the inspired individual, apparently working alone, emerging from the laboratory with a new discovery.

Unfortunately, however, there is many a step between the blush of first discovery (plus analysis of its significance) and an understanding of the causes involved. In the 51 years that have passed since Dr. Ravenal reported the results of his work to the Philadelphia Pathological Society, many workers have attempted to gain an understanding of the phenomenon he described—that certain bacteria can be cooled to extremely low temperatures yet still survive. As time has passed, the techniques applied have become more complex and the equipment more elaborate and expensive. The truth of the matter is that first discoveries may often be the fruit of the lone investigator working with a few test tubes, but the quantitative study of such a discovery, the analysis of the significance of the variables involved—in short, the translation of a brilliant idea into useful knowledge—require a great deal more effort and, usually, a great deal more equipment.

During the first three decades of this century, even more confusing results were obtained than previously. Some workers reported that all (or almost all) of certain bacterial populations were destroyed when stored at temperatures of −2° to −17° C., whereas other researchers reported only a slight reduction in population after storage under these same conditions of temperature. A few observers performed experiments which indicated that these differences might be due to a protective influence of the medium in which the bacteria were cooled. Even more confusing were the results obtained when bacterial suspensions of the same type were cooled rapidly to very low temperatures. In most cases it was found, as by Ravenal, that the organisms survived such treatment, but that the majority of them died when they were stored at temperatures between −2° and −20° C.

ZONE OF CRYSTALLIZATION CONCEPT

For some time these contradictory observations were considered as accidental happenings and were probably attributed by various workers to variations in techniques. However, in the middle twenties, several histologists, notably Stiles in England and Plank in Germany, published articles concerning the temperature "zone of crystallization," observed in the freezing of muscle fibers. They reported that the majority of the damage incurred by the muscle fiber during freezing took place when ice crystals began to form within and around the fibers, and they referred to the temperature range in which this occurred as the "zone of crystallization." Although there does not seem to have been much free communication between those interested in the effect of low temperatures on microorganisms and these histologists, nevertheless by the latter part of the third decade some consideration was given the role that ice formation might play in causing death at low temperatures.

The theories that resulted have been found to be fully as conflicting as the observations which they attempt to explain. As diverse as these theories were, none made any real effort to explain why it was that certain bacterial populations would diminish or even disappear at temperatures between −2° and −20° C., although those identical populations could be cooled as low as −253° C. without significant injury.

VITRIFICATION CONCEPT

In 1937, however, B. J. Luyet of St. Louis University introduced a new concept

—that living matter could be vitrified by exposure to extremely low temperatures, and that vitrified protoplasm could then be warmed to room temperature without suffering injury. The concept of the vitreous state is not new—silicate glasses are hundreds of years old—but a wider application than in glasses was not considered until 1898 when Tamman pointed out that a large number of substances can be obtained as glasses and suggested that this property might be universal.

Perhaps a few words on the vitreous state might be in order at this point. In passing from the liquid to the solid state, most substances become crystalline. If, however, the tendency for centers of crystallization to form in a substance is of a low order, or if the cooling from liquid to solid state is accomplished too quickly for crystallization to occur, the resultant solid will be glassy, or vitreous. Some workers prefer to think of the vitreous state as liquid rather than solid because, in the molecular sense, no orientation into crystalline form has occurred. In any event, a vitrified or glassy material has the property of transparency, whereas the crystalline form of the same substance is opaque or translucent.

Because we are all familiar with ordinary glass, I believe that we can readily appreciate the concept of vitrification and devitrification. Thus, when a molten mix of glass is cooled rapidly enough below its melting point, it becomes a transparent solid; if, however, the mix is held at a temperature just below its melting point for a sufficient length of time, crystallization occurs. However, once a vitrified solid is cooled well below the melting point, and kept there, it will remain glassy or vitreous for very long periods of time; perhaps indefinitely. On the other hand, if a vitrified glass is warmed to a temperature near its melting point, it will crystallize, or devitrify, becoming opaque or translucent. Most of you have probably observed this phenomenon in your attempts to work glass and can recall the unsightly cloudy appearance of some of your early bends or seals. This cloudy appearance was due to crystal formation within the glass when it was held too long at a temperature just below its melting point.

As has been indicated earlier, B. J. Luyet was the first to publish work indicating that living matter might be vitrified. Alexander Goetz and Scott-Goetz apparently arrived at this conclusion independently, predicting that vitrification would not be harmful to protoplasm because of the fact that crystal orientation does not take place in the vitreous state, and therefore vitrification would cause no change. At any rate, Luyet and Goetz have presented a partial explanation of why certain bacteria can withstand temperatures as low as −253° C., but are unable to survive for any appreciable length of time at temperatures in the range of zero to −20° C. Their theory postulates that when the temperature of the organism is lowered at a sufficiently rapid rate, the water within the protoplasm is transferred from a liquid or gel state to a vitreous state, without change, no crystals being formed. The rate of cooling required to induce vitrification in protoplasm is of the order of 1000° C. per second, and the rate of warming from the vitreous state must be of the same order of magnitude, otherwise devitrification occurs — that is, the vitreous matter will become crystalline prior to melting, and, if crystallization occurs, death follows in most instances.

Thus the apparent anomaly of the discontinuity of the effect of lowering the temperature of microorganisms has been partially explained. The full significance of this explanation, however, has not yet been elucidated, and many inconsistencies remain to be resolved. For example, many microorganisms can not only live, but can actually multiply in the temperature range of zero to −15° C., when, in the light of the theory just given, it would appear that crystallization and death should occur under these conditions.

FUTURE WORK

It is our belief that a great deal of information as to the exact effect of temperature on microorganisms must be obtained before we can really understand the phenomenon of life and death at low temperatures. It may well be that many of the apparently contradictory observations can be correlated if we can evaluate the variables

involved in experiments dealing with the survival of microorganisms at low temperatures. The environment, type and age of the cells, rate of cooling and thawing, the water content of the cells themselves, and many other factors may have bearing on these phenomena.

However, it seemed to us that the effect of temperature itself must be evaluated quantitatively before we can progress further toward an understanding of the other factors involved. For this reason, we are now engaged at Georgia Tech in just such a study—on the effect of low temperatures on the survival of microorganisms. This work has been made possible by a grant-in-aid from the National Institutes of Health.

Incidentally, the securing of this grant is illustrative of some of the problems that confront science when it seeks funds. The public health people are interested in the possible survival of pathogenic bacteria in frozen foods as a potential public health hazard. We, on the other hand, are primarily interested in securing further information as to the mechanics of life and death at low temperatures, but had no funds available for this work. The Institutes of Health does have funds for research on problems of importance to it; we therefore presented to the Institutes a proposal for work which would yield the information they desire and which would at the same time further the development of our own broad program on the preservation of foods by freezing.

In carrying out this particular project, our approach is radically different from that of Ravenal, who recorded the first complete experiments on the survival of microorganisms at extremely low temperatures. His experiments were qualitative in nature; they proved conclusively that certain pathogenic bacteria can withstand the low temperatures employed. In order to understand the full significance of this observation, however, it seems necessary to carry out quantitative experiments to determine whether there are degrees of response to cold, or whether it is an "all or none" effect. Ravenal's equipment was simple and inexpensive, but, because we must attempt to disassociate the organism as nearly as possible from the influence of its environment in order to study the effect of temperature *per se*, our equipment has to be more elaborate and more expensive. Ravenal probably worked alone, but completely quantitative studies of the effect of low temperatures upon life require the joint application of several sciences; therefore, we have attacked our problems on a project basis, consulting not only with biologists and physicists, but also with chemists and engineers. Such are the differences between qualitative discoveries and the quantitative investigations based on these discoveries.

It may appear to many that it is "a long way" from the freezing of bacteria to the freezing preservation of foods. That is quite correct, but certain of the fundamental changes that occur during exposure to low temperatures must be common to all protoplasm, so that, if we are ever able to understand these changes, we will then have taken a great step forward in the systematic study of preservation. As was indicated earlier, our food preservation research at Georgia Tech has been based on the theory that fundamental studies on processes and causes will yield information which can be applied to practical considerations, and that such a long-range program will produce more practical and useful results in a shorter length of time than will short-term, so-called applied research.

In carrying on our investigations we have delved into many apparently unrelated fields, but the data obtained and the hypotheses derived from the data have already proved of value in predicting the probable operation of several processes which are of practical significance in the commercial freezing preservation of foods.

In the interpretation and correlation of the results of a broad program, there is no known method of predicting in advance the particular field of investigation or source from which the most valuable information may be obtained—as was discovered by a little old lady at church. After an unusually long sermon, the preacher was pleasantly surprised by her somewhat effusive compliments on his discourse, the theme of her remarks being how greatly his sermon had helped her. When he inquired as to the

particular portion of the sermon that had aided her the most, she replied, "Why, when you were talking about the development of different personalities, you said, 'Just as the fuchsia requires shade, and the poppy flourishes in full sun.' Well, until then I never understood why Mrs. Jones' fuchsia were always prettier than mine."

EXPOSURE TESTING

Continued from Page 8

complete evaluation, faulty techniques cannot be detected early. Therefore, painstaking care must be exercised in the planning and execution of testing procedure so that the exposure data obtained may be accurately representative of the particular conditions specified. Obviously, test results can have little meaning unless these conditions are well defined and all variables are closely controlled.

Many investigators have published articles and papers on exposure testing and test results.[2 3 4 5 6 7 8 9] Unfortunately, the results of different investigators have often proved difficult to correlate because of variations in experimental conditions. Nevertheless, all available information should be evaluated and utilized as fully as possible before setting up a testing program. While some principles are generally applicable and a few techniques have become well standardized, the advice of experienced paint technologists is helpful in selecting the most appropriate procedures and conditions for a particular program.

A complete description of details of all the various exposure testing techniques is beyond the scope of this article. However, description of the methods now being used in a testing program at Georgia Tech will indicate some of the considerations involved.

Georgia Tech Program

The coatings laboratory at the Georgia Tech Engineering Experiment Station and the research programs on exterior house paints have been described in a previous article.[10] The Station, in cooperation with the Southern Paint and Varnish Production Club and the Southern Pine Association, is conducting a comparative study of what are considered to be the best exterior primer-top coat systems available today for application to southern yellow pine. Before the laboratory work on this project was begun, the pertinent literature on the subject was reviewed and a program outline prepared along lines suggested in staff consultations and in correspondence with many leading paint technologists.

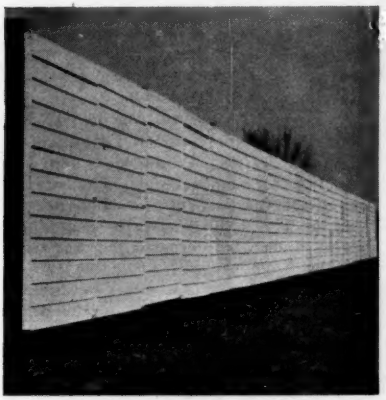
For any paint exposure testing program the conditions and variables that require definition and control may be grouped into eight types as indicated by the headings in the discussion that follows. Under these headings the test methods selected for the Georgia Tech program are described, together with a discussion of some of the reasons for selection of particular methods.

Chemical Composition of Paints

The ten primer paints and 15 top-coat paints that are being studied represent formulations that, for the most part, are familiar to the trade. A few are variations of these familiar formulations. The paint materials used in preparing these paints were all commercially available brands of the types normally used in paint manufacture. Where it was possible, a single brand and lot of material was used throughout all the formulations. For example, single lots of magnesium silicate extender pigment, raw linseed oil and mineral spirits were used in every paint. The same principle was applied to the maximum extent possible with all the other paint materials required. The purpose of this procedure was to eliminate the possibility of having variable properties in any of the materials used in these paints.

Preparation Methods

Approximately one gallon of each paint was prepared. The required amounts of pigment and vehicle materials were carefully weighed out, and the pigment was charged into a laboratory paste mixer. A sufficient amount of vehicle was added to produce a thick paste which was mixed until it became quite "smooth." Then more vehicle was added and the paste was further dispersed on a three-roll mill adjusted to produce a grind of about 2.5 (North Standards Scale) in a single pass. Finally the remaining vehicle was added and the paint thinned to satisfactory consistency and packaged in one-gallon cans.



This angle view of a 50-panel test fence accentuates the differences in appearance between the various paint systems before weathering.

While several different types and combinations of pigment-dispersing equipment are used by manufacturers in preparing house paints, the paste mixer and three-roll mill combination was selected for the present work because it is one most frequently employed. Other popular types of equipment include the ball mill and, more recently, the paste mixer-colloid mill combination.

Physical Properties of Paint

Both chemical and physical data on paint materials were supplied by materials manufacturers. Physical properties which required experimental determination and adjustment were the fineness of grind (degree of dispersion of pigment in vehicle), the weight per gallon of paint and its consistency. These properties were determined by means of the Hegman Gage, a weight-per-gallon cup and the Modified Stormer Viscometer, respectively. The use of these control instruments has become fairly standard in the industry. Fineness of grind and consistency were determined and controlled because these variables affect the properties of the applied paint film. Determination of the weight-per-gallon of the finished paint allowed a rough check on the accuracy of the composition.

Test Panels

Single primer-top coat systems were applied to panels consisting of six boards of selected No. 105 pattern southern yellow pine siding, six inches wide and four feet long. These boards were mounted together on a frame so that a test area of about three feet by four feet was provided. A one-foot length along the left side of the front of each board was reserved for application of a standard or reference primer and top coat; the remaining area, about nine square feet, was used for the experimental system. The back side of each board was given a heavy coat of aluminum primer to reduce the absorption of moisture, thereby simulating, to some extent, the protection normally given siding by the building. Moisture content of the wood was required to be 12 ± 1 per cent at the time of application of the test primer, and, as determined by the Kaydel Gage, it was very nearly 12.0 per cent in most cases. After all paints were applied and allowed to dry, the panels were mounted on a test fence.

The reasons for evaluating these paints on southern yellow pine lumber have been discussed previously.[10] The No. 105 pattern of siding is a popular type. The comparatively large test panel and the use of a standard system on each board were safeguards employed to reduce the possibility of faulty interpretations resulting from nonuniformity of the wood structure. Moisture content, while not a critical variable at the levels encountered,[11] was controlled mainly to preclude the possibility of painting damp panels.

Application Methods and Spreading Rate

All paints were applied indoors in a well ventilated room and allowed to dry for two days between coats and an additional two days before mounting on the test fence. A 2½-inch hog bristle brush was used for spreading the paints. A dry film of constant average thickness was achieved on all panels by applying a calculated volume of paint to each panel from a calibrated surgical syringe and then brushing this paint to a uniform coating. The calculations and assumptions for determining dry film thickness for a given spreading rate were developed by Browne,[12] and further details

of the method used have been described by Wilkins and Weber.[3]

Paint films may be evaluated on the basis of either constant wet-film thickness or constant dry-film thickness. The former method is more appropriate when one is interested merely in the comparative performance of each of several different proprietary paints applied to a given surface area. However, in the present work interest was directed toward variations in the nonvolatile, film-forming ingredients in paint formulations. For this purpose constant average dry-film thicknesses are desirable for comparative evaluations.

Direction and Angle of Panel Exposure

Panels for the Georgia Tech program were exposed at a test station in a rural area about 10 miles north of Atlanta. All panels were exposed vertically, facing due south. South exposure of vertical test panels is generally considered to test most accurately the ultimate performance of a house paint, since initial paint film failure and the most severe weathering effects usually occur on the south side of a house.

Paint technologists have observed that the changes occurring in paint films exposed on test fences are not always exactly identical to the changes in the same films applied to dwellings. In heated or air-conditioned buildings particularly, large temperature and moisture gradients and often higher moisture content may be found in the siding than in test fence panels.[10] These effects influence the durability of a paint film; however, in moderate climates test fence data correspond very closely to actual tests on properly constructed dwellings. The obvious difficulties in controlling conditions for "practical" tests on houses will usually rule out this method when a comparative study of a large number of paint systems is necessary as in the present investigation.

Standards for Comparison and Evaluation

It was mentioned previously that a one-foot length of each board in these tests was reserved for a "standard" or "control" paint system. In addition to supplying a "frame of reference" for evaluating the performance of the experimental systems, this control paint system also serves as a monitor on each board to detect erratic wood behavior which often occurs in yellow pine lumber. The control paint system has been previously tested on southern yellow pine, and its performance is well known.

An *Exposure Standards Manual* has been obtained from the National Federation of Paint and Varnish Production Clubs for evaluation of paint failures. This manual contains photographs illustrating the various types of film failures and the progressive degrees of such failures, assigning numerical values of from 10 to 0 (10 being perfect, 0 being the poorest conceivable condition). These photographs are being used to define the condition of the paint films at various intervals during exposure.

Recording of Data

Test panels are checked for loss of gloss and premature chalking at the end of one month. Thereafter the panels are checked once every three months, and the condition of each system is recorded on an exposure chart, together with the condition of the control system on each panel. Thus the performance of each paint system is followed and described and compared to the performance of a well known control system during the process of normal weathering.

Interpretation of Test Results

The foregoing description of test methods has indicated the multiplicity of variables involved in any exposure testing program. The investigator will usually prefer to "let the results speak for themselves" rather than attempt to draw any broad conclusions from a single series of studies. The pitfalls of attempting to extrapolate empirical relations beyond the range of experimental data have been disastrously illustrated on many occasions. While the investigator may have full confidence in conclusions that fall within the scope of his investigation, attempts to apply these conclusions to other sets of conditions can only be regarded at best as "educated guessing."

It becomes clear, then, why apparently contradictory results have sometimes been reported of exposure studies by different investigators. Usually these results were

Continued on Page 23

SUBJECT AND AUTHOR INDEX

MAY, 1950-MARCH, 1951

A

2-Acetyl thiophene, tertiary alcohol derivatives, Ja '51, 15
A-C network calculator, S '50, 18
Adsorption, see Dye adsorption
Aeronautical engineering,
———, aerodynamic equilibrium equation of the lifting rotor, S '50, 22
———, airfoil studies, by hydraulic analogy, S '50, 16
——— ———, in compressible flow, S '50, 21
———, airplane transfer functions, determination and classification, S '50, 22
———, digests of graduate theses, S '50, 21
———, research, S '50, 16
Aerosols, settling rate of particles, S '50, 12
Allis-Chalmers solvent extraction unit, Ja '51, 19, 22, 23
Aluminum alloy sheet,
———, fatigue and corrosion fatigue study, S '50, 21
———, flexure fatigue, S '50, 21
Anderson solvent extraction unit, see also Exsolex, Ja '51, 19, 21
Aptitude tests, S '50, 19
Army reserve research and development program, My '50, 9
Assembling machine, automatic, S '50, 16
Athey, Robert J.,
———, the absorption spectrum of nitryl chloride, Ja '51, 15
Aviation clock dial, My '50, 13
Azeotrope in the bromine-carbon tetrachloride system, Ja '51, 16

B

Bartholomay, Henry William,
———, solubilities in the system gallium-mercury, Ja '51, 15
Bartlett's Ferry Dam, see Dam
Battery, fundamental studies, S '50, 11
Beaver, William Morrisey,
———, tertiary alcohol derivatives of 2-acetyl thiophene, Ja '51, 15
Bergsman microhardness tester, My '50, 4
Beryllium, analytical investigation, S '50, 12
Biochemistry,
———, microorganism survival at low temperatures, S '50, 14
———, research, S '50, 14
———, water and sewage analysis, S '50, 14
Bizzell, Oscar M.,
———, planning a small radioisotope program, N '50, 9
Blaw-Knox solvent extraction unit, Ja '51, 20, 22
Bodiford, Robert Graham,
———, an investigation of the effect of surface finish on the flexure fatigue strength of 75S-T6 aluminum alloy sheet, S '50, 21
Bollinger, Everett, Jr.,
———, capital issues and their cyclical implications, My '50, 11
Bollman solvent extraction unit, Ja '51, 19
Bonotto solvent extraction unit, Ja '51, 19
Brawley, Bolling Gay,
———, an investigation of capillary spreading power of emulsions, N '50, 17
Brick masonry, reinforced, S '50, 16
Bromine-carbon tetrachloride system, azeotrope, Ja '51, 16
Burrows, W. H.,
———, graphical and mechanical methods of computation, My '50, 5

C

Calculator, A-C network, S '50, 18
Capillary spreading power of emulsions, N '50, 17
Capital issues, cyclical implications, My '50, 11
Carbon monoxide from gas heaters, S '50, 12
Carbon tetrachloride,
———, -bromine system, azeotrope, Ja '51, 16
———, -toluene rectification, N '50, 17
Carboxylic acid, 2-thiophene, preparation of esters, Ja '51, 16
Carter, William L.,
———, characteristics of a packed distillation column: correlation of H.T.U. with operating variables for rectification of carbon tetrachloride-toluene mixtures, N '50, 17
Catchpole, Eric John,
———, application of the hydraulic analogy to study the performance of two airfoils in compressible flow, S '50, 21
Cattle-weighing scale, S '50, 15
Cement, effect of additives on the physical properties, N '50, 18
Cerium, relations of temperature, crystal structure and superconductivity, My '50, 12
Chemical engineering, digests of graduate theses, N '50, 17
Chemical Sciences Division, S '50, 11
———, new head, My '50, 24
Chemistry,
———, digests of graduate theses, Ja '51, 15
———, research, S '50, 11
Cheng, Fa-wu,
———, the extraction of oil and protein from cottonseed by a water-grinding and centrifuging process, Ja '51, 16
Chromium determination in water and sewage, S '50, 14
Circuits, high-frequency, crystal-controlled oscillators, S '50, 18
Civil engineering research, S '50, 16
Cleaning farmers' stock peanuts, Ja '51, 3
Cliett, Charles Buren,
———, a study of fatigue and corrosion fatigue for 24ST aluminum alloy sheet, S '50, 21
Clock dial, aviation, My '50, 13
Coatings,
———, facilities for study, N '50, 20
———, flooring materials, S '50, 14
———, paints, S '50, 13
———, plastics, rubber and resins, N '50, 4
———, research, S '50, 13; N '50, 3
———, varnishes and resins, S '50, 14
Cofield, E. P., Jr.,
———, solvent extraction—a new method in an old industry, Ja '51, 5
Colburn equation, applicability to carbon tetrachloride-toluene solutions, N '50, 17
Compton, Philip Robinson,
———, an analytical procedure for determination of lift and drag forces for bodies of revolution, S '50, 22
Computation, graphical and mechanical methods, My '50, 5; S '50, 18
Copper spark method of spectrographic analysis, Ja '51, 16
Corrosion fatigue study of aluminum alloy sheet, S '50, 21
Cottonseed,
———, oil and protein extraction by water-grinding and centrifuging, Ja '51, 16
———, solvent extraction, Ja '51, 5, 6, 21, 23

Cottrell, Bert M., Jr.,
———, the preparation of certain esters of 2-thiophene carboxylic acid, Ja '51, 16
Cox, Fred W., Jr.,
———, the State Engineering Experiment Station, 1949-1950, S '50, 7
Crosland, Dorothy M.,
———, the Georgia Tech Library as a tool for science and industry, N '50, 5
Crown solvent extraction unit, Ja '51, 20, 23
Crystallization, zone of, Mr '51, 13
Crystals,
———, high-frequency oscillator circuits, S '50, 18
———, quartz, impedance-frequency charactristics, My '50, 12

D

Dam,
———, Bartlett's Ferry, description, Ja '51, 7
———, ———, engineering investigation, Ja '51, 7
———, ———, hydraulic model tests, Ja '51, 10
———, ———, modifications, Ja '51, 7
———, chute spillway, S '50, 16
———, erosion reduction, S '50, 16
Detrex solvent extraction unit, Ja '51, 20, 23
Dielectric and reflecting properties of sea water at microwave frequencies, S '50, 18
Distillation,
———, applicability of Colburn equation to carbon tetrachloride-toluene, N '50, 17
———, packed column characteristics, N '50, 17
———, rate of molecular, N '50, 18
Dixon, Frederick,
———, the impedance-frequency characteristics of quartz crystals, My '50, 12
Drag and lift forces on bodies of revolution, prediction, S '50, 22
Dwyer, Joseph S.,
———, an analysis of motion and time study training as given by colleges and industry, My '50, 11
Dye adsorption applied to fine particles, S '50, 12

E

Education and research, N '50, 2
Electromagnetic waves,
———, dielectric and reflecting properties of sea water at microwave frequencies, S '50, 18
———, propagation studies, S '50, 17
Electronics research, S '50, 17
Electron Microscope, Mr '51, 3
———, calibration, Mr '51, 11
———, description and operation, Mr '51, 4
———, magnification, Mr '51, 4
———, replica techniques, Mr '51, 11
———, shadow-casting techniques, Mr '51, 11
———, specimen limitations, Mr '51, 10
———, studies, S '50, 18
Elliott, Thomas A.,
———, cleaning farmers' stock peanuts, Ja '51, 3
Emulsions, capillary spreading power, N '50, 17
Engineering Experiment Station, see State Engineering Experiment Station
Engine, gasoline, development, S '50, 15
Erosion, corrective measures applied to dam apron, S '50, 16
Experiment Station, see State Engineering Experiment Station
Exposure,
———, station, N '50, 20
———, testing of paints, N '50, 3; Mr '51, 7
———, ———, accelerated tests, Mr '51, 23
———, ———, Georgia Tech program, Mr '51, 16
———, ———, interpretation of results, Mr '51, 18
———, ———, techniques, Mr '51, 8
Exsolex solvent extraction unit, see also Anderson, Ja '51, 19, 21, 22, 23
Extraction, see also Solvent extraction,
———, mechanical, Ja '51, 6
———, solvent, Ja '51, 5
———, water-grinding and centrifuging, S '50, 12; Ja '51, 16

F

Fatigue,
———, and corrosion fatigue study of aluminum alloy sheet, S '50, 21
———, flexure tests of aluminum alloy sheet, S '50, 21
Fine particles, see also Micromeritics, collection of radioactive dusts, N '50, 15
Flexure fatigue tests of aluminum alloy sheet, S '50, 21
Flooring materials, S '50, 14
Floyd, Acey Lee Roy, Jr.,
———, effect of temperature on the crystal structure of lanthanum, cerium, neodymium, and praseodymium. The relation between crystal structure and superconductivity, My '50, 12
Fluoride determination in water and sewage, S '50, 14
Foods,
———, freezing, S '50, 12; Mr '51, 5
———, ———, changes in physical characteristics, S '50, 13
———, research, S '50, 12
Freezing,
———, of foods, Mr '51, 5
———, of highly organized bodies, Mr '51, 6
———, of microorganisms, Mr '51, 12
———, vitrification, Mr '51, 13
———, zone of crystallization, Mr '51, 13
French solvent extraction unit, Ja '51, 20, 22, 23

G

Gallium-mercury solubilities, Ja '51, 15
Gas heaters, production of carbon monoxide, S '50, 12
Gasoline engine development, S '50, 15
Georgia Institute of Technology, see also State Engineering Experiment Station
———, Army reserve research, My '50, 9
———, coatings research, N '50, 4
———, ———, facilities, N '50, 20
———, electron miscroscope, Mr '51, 3
———, frozen foods, Mr '51, 5
———, hydraulic weighing scale, N '50, 7
———, library, N '50, 5
———, metallurgical research, My '50, 3
———, paint exposure-test program, Mr '51, 16
———, research on peanut cleaning, Ja '51, 3
Georgia, research for, S '50, 9
Government-financed research, Ja '51, 2
Graphical and mechanical methods of computation, My '50, 5; S '50, 18

H

Hall, R. A.,
———, hydraulic weighing scale, N '50, 7
Harrison, E. S.,
———, modifications of dam checked by hydraulic models, Ja '51, 7
Health physics in radioisotope laboratories, N '50, 14
Hearing and vision tests in employee selection, S '50, 20
Helicopter rotor, effect of twist and taper of the blades in vertical descent, S '50, 16
High-frequency crystal-controlled oscillator circuits, S '50, 18

Hildebrandt solvent extraction unit, Ja '51, 20, 23
Honnell, M. A.,
———, television today, S '50, 3
Hoover, William Stough, Jr.,
———, the effect of certain additives upon the physical properties of Portland cement, N '50, 18
Hydraulic,
———, analogy, applied to airfoils, S '50, 16
———, ———, ———, in compressible flow, S '50, 21
———, ———, applied to problems of supersonic wind tunnel design, S '50, 22
———, model check of dam modifications, Ja '51, 7
———, research, S '50, 16
———, weighing scale, N '50, 7

I

Industrial engineering, digests of graduate theses, My '50, 11
Industrial management, digests of graduate theses, My '50, 11
Industrialization of the South, trained manpower from Georgia Tech, Mr '51, 2
Information service, technical, at Georgia Tech, N '50, 21
Institute of Nuclear Studies, N '50, 10
Interviews, applied to employment problems, S '50, 24

J

Janus, Sidney Q.,
———, psychological tools for business and industry, S '50, 19
Jones, W. Dale,
———, wage incentive payment for multiple-machine assignments, My '50, 11

K

Kennedy solvent extraction unit, Ja '51, 20, 22, 23
Kethley, T. W.,
———, life and death at low temperatures, Mr '51, 5
Kindsvater, C. E.,
———, modifications of dam checked by hydraulic models, Ja '51, 7
Kruger, Jerome,
———, an azeotrope in the system bromine-carbon tetrachloride, Ja '51, 16

L

Lanthanum,
———, relations of temperature, crystal structure and superconductivity, My '50, 12
———, superconductivity, S '50, 11
Lefevre and Tollens method of uronic acid analysis, applied to polyanhydrouronic acids, N '50, 18
Library, Georgia Tech, N '50, 5
———, collections, N '50, 6
———, facilities, N '50, 6
Life and death at low temperatures, Mr '51, 5
Lift and drag forces on bodies of revolution, prediction, S '50, 22
Lift coefficient, maximum for swept-back wings, S '50, 16
Little, Albert P.,
———, solvent extraction of Georgia pine needles, N '50, 18
Low temperatures, see also Very low temperature,
———, effect on the survival of microorganisms, S '50, 14
———, life and death at low temperatures, Mr '51, 5

M

Manganese dioxide, electrochemical synthesis, for batteries, S '50, 11
Masonry, reinforced brick, S '50, 16
Mathematical research, S '50, 18
Mechanical engineering research, S '50, 14
Mechanical Sciences Division, S '50, 14
Medical equipment, S '50, 16
Meek, Richard Lee,
———, rate studies in molecular distillation, N '50, 18
Mercury-gallium solubilities, Ja '51, 15
Metallurgy,
———, equipment, My '50, 4
———, research at Georgia Tech, My '50, 3
Microhardness tester, My '50, 4
Micromeritics, see also Fine particles,
———, settling rate of particles in aerosols, S '50, 12
———, surface properties of fine particles, S '50, 11
Microorganisms, survival at low temperatures, S '50, 14; Mr '51, 12
Microwaves,
———, dielectric and reflecting properties of sea water, S '50, 18
———, propagation, S '50, 17
Military research, S '50, 2
———, Army reserve, My '50, 9
Modulation, velocity, S '50, 18
Molecular distillation, rate, N '50, 18
Morris, John,
———, the effect of oxidation on the 2,3-positions on the decarboxylation of certain polyanhydrouronic acids, N '50, 18

N

Neodymium,
———, relations of temperature, crystal structure and superconductivity, My '50, 12
———, superconductivity, S '50, 11
New, Noah Carroll,
———, the aerodynamic equilibrium equations of the lifting rotor, S '50, 22
Ninety-degree phase difference systems, S '50, 18
Nitryl chloride, absorption spectrum, Ja '51, 15
Nomographs, My '50, 8; S '50, 18

O

Oak Ridge Institute of Nuclear Studies, N '50, 10
Oilseeds,
———, extraction, S '50, 12
———, solvent extraction, Ja '51, 5, 21
Oscillator circuits, high-frequency, crystal-controlled, S '50, 18

P

Paints,
———, exposure testing, N '50, 3; Mr '51, 7
———, exterior, present development, Mr '51, 7
———, studies of systems on southern yellow pine, S '50, 13
Particle size distribution, determination by settling rate, S '50, 12
Peanuts,
———, cleaning, Ja '51, 3
———, ———, methods, Ja '51, 4
———, ———, ———, electrostatic, Ja '51, 4
———, ———, ———, mechanical, Ja '51, 17
———, ———, ———, washing and drying, Ja '51, 4
———, planting and harvesting machinery, S '50, 15
———, sampling, cleaning, handling and shelling, S '50, 14
———, solvent extraction, Ja '51, 5, 6, 21, 22, 23

Phase difference systems, ninety-degree, S '50, 18
Physics, digests of graduate theses, My '50, 12
Physics Division, S '50, 17
Pine needles, solvent extraction, N '50, 18
Pine, southern yellow, painting, Mr '51, 16, 17
Plastics, use as coating, N '50, 4
Plourd, Webster William,
———, aviation clock dial characteristics in relation to speed and accuracy of reading, My '50, 13
Polyanhydrouronic acids, decarboxylation, N '50, 18
Portland cement, effect of certain additives on the physical properties, N '50, 18
Power transmission, A-C network calculator, S '50, 18
Praseodymium,
———, relations of temperature, crystal structure and superconductivity, My '50, 12
———, superconductivity, S '50, 11
Propagation of electromagnetic waves, S '50, 17
Psychological tests,
———, aptitude, S '50, 19
———, hearing, S '50, 20
———, limitations, S '50, 19
———, vision, S '50, 20
Psychological tools for business and industry, S '50, 19
Publications,
———, of the State Engineering Experiment Station, S '50, 10
———, research, My '50, 2

Q

Quantitative research, Mr '51, 2
Quebracho, red, effect on the physical properties of cement, N '50, 18

R

Radar research, S '50, 17
Radioactive wastes disposal, N '50, 14
———, concentration of radioactivity, N '50, 15
———, dust collection methods, N '50, 15
———, sewage system contamination, N '50, 15
Radioisotopes, planning for a research program, N '50, 9
———, cost, N '50, 11
———, equipment, N '50, 12
———, health physics, N '50, 14
———, laboratory, N '50, 11
———, literature available, N '50, 10
———, training necessary, N '50, 10
———, waste disposal, N '50, 14
Rare earths, relations of temperature, crystal structure and superconductivity, My '50, 12
Raudebaugh, R. J.,
———, metallurgical research at Georgia Tech, My '50, 3
Reflecting and dielectric properties of sea water at microwave frequencies, S '50, 18
Reid, George W.,
———, planning a small radioisotope program, N '50, 9
Replica techniques in electron microscopy, Mr '51, 11
Research,
———, aeronautical, S '50, 16
———, Army reserve program, My '50, 9
———, biochemical, S '50, 14
———, chemical, S '50, 11
———, civil engineering, S '50, 16
———, coatings, S '50, 13; N '50, 3
———, education's relation to, N '50, 2
———, electronics, S '50, 17
———, engineering schools' contributions, Ja '51, 2
———, food, S '50, 12
———, for Georgia, S '50, 9
———, future of, S '50, 2
———, government-financed, Ja '51, 2
———, hydraulics, S '50, 16
———, industrial and governmental, My '50, 2
———, mathematical, S '50, 18
———, mechanical engineering, S '50, 14
———, metallurgical, at Georgia Tech, My '50, 3
———, military, S '50, 2
———, need for facilities, N '50, 2
———, nonprofit, Mr '51, 24
———, publications, My '50, 2
———, quantitative, Mr '51, 2
———, radar, S '50, 17
———, television, S '50, 18
———, textile, S '50, 14
———, wood, S '50, 14
Research Engineer, editor, Ja '51, 24
Resins in paints and coatings, S '50, 14
Rosselot, Gerald A.,
———, the State Engineering Experiment Station, 1949-1950, S '50, 7
Rotocel, see Blaw-Knox solvent extraction unit
Rotor, lifting, aerodynamic equilibrium equations, S '50, 22
Rowlock brick masonry, S '50, 16
Rubber, use as coating, N '50, 4

S

Safety engineering, digests of graduate theses, My '50, 13
Scale, hydraulic weighing, S '50, 15; N '50, 7
———, mechanism, N '50, 8
———, operation, N '50, 23
Seacord, Charles L., Jr.,
———, determination and classification of airplane transfer functions, S '50, 22
Sea water, dielectric and reflecting properties at microwave frequencies, S '50, 18
Sessions Brothers' solvent extraction unit, Ja '50, 21, 22
Settling rate measurement, S '50, 12
Sewage,
———, and water analysis, S '50, 14
———, sulfate determination, S '50, 14
———, systems, effect of radioactivity on, N '50, 15
Shadow casting techniques in electron microscopy, Mr '51, 11
Sherwin-Williams solvent extraction unit, Ja '51, 20, 22, 23
Slide rules, My '50, 5
Sodium pyrophosphate, effect on the physical properties of cement, N '50, 18
Solubilities in gallium-mercury system, Ja '51, 15
Solvent extraction, see also extraction,
———, oilseeds, Ja '51, 5
———, ———, economic aspects, Ja '51, 23
———, ———, forepressing, Ja '51, 6
———, ———, future trends, Ja '51, 23
———, ———, history, Ja '51, 5
———, ———, meal and solvent separation, Ja '51, 21
———, ———, oil and solvent separation, Ja '51, 21
———, ———, units, Ja '51, 19
———, pine needles, N '50, 18
Soo, Shao-Lee,
———, a study of the effect of lubrication on the dynamics of spinning spindles, My '50, 13
Soybeans, solvent extraction, Ja '51, 6, 18, 21, 23
Spectrographic analysis, copper spark method, Ja '51, 16
Spectrum, nitryl chloride, Ja '51, 15
Spillway, see Dam
Spinning spindles, effect of lubrication on dynamics, My '50, 13
State Engineering Experiment Station, see also Georgia Institute of Technology
———, 1949-1950, S '50, 7
———, Chemical Sciences Division, My '50, 24
———, purpose, N '50, 21

———, Technical Information Service, N '50, 21; Ja '51, 24
Sulfate determination in water and sewage, S '50, 14
Supersonic wind tunnel design problems, S '50, 22
Surface properties of fine particles, S '50, 11

T

Technical Information Service,
———, at Georgia Tech, N '50, 21
———, head, Ja '51, 24
Television,
———, today, S '50, 3
———, ———, color, S '50, 5
———, ———, industrial application, S '50, 6
———, ———, legal aspects, S '50, 4
———, ———, technical aspects, S '50, 5
———, velocity modulation, S '50, 18
Textiles,
———, cotton-card fly, reclamation, S '50, 14
———, engineering, digests of graduate theses, My '50, 13
———, fabric analysis, S '50, 14
———, machinery, pickerstick check strap study, S '50, 14
———, research, S '50, 14
———, yarns, physical study, S '50, 14
Theses, digests of graduate,
———, aeronautical engineering, S '50, 21
———, chemical engineering, N '50, 17
———, chemistry, Ja '51, 15
———, industrial engineering, My '50, 11
———, industrial management, My '50, 11
———, physics, My '50, 12
———, safety engineering, My '50, 13
———, textile enginering, My '50, 13
Thioisophthalaldehyde condensation, Ja '51, 17
Thiophene, 2-acetyl, tertiary alcohol derivatives, Ja '51, 15
2-Thiophene carboxylic acid esters, preparation, Ja '51, 16
Thomas, Gerald B.,
———, application of water channel compressible gas analogies to problems of supersonic wind tunnel design, S '50, 22
Time and motion study, training, My '50, 11
Titanium, My '50, 14
———, -copper system, My '50, 15
———, alloys, S '50, 12
Tollens and Lefevre method of uronic acid analysis, applied to polyanhydrouronic acid, N '50, 18
Toluene-carbon tetrachloride rectification, N '50, 17
Tooke, W. R., Jr.
———, coatings research at Georgia Tech, N '50, 3
———, exposure testing—a guide for improvement of exterior paints, Mr '51, 7
Transfer functions, airplane, determination and classification, S '50, 22
Tung nuts, solvent extraction, Ja '51, 6, 23

U

Uronic acid analysis, Lefevre and Tollens method applied to polyanhydrouronic acid, N '50, 18

V

Van Leer, Blake R.,
———, education and research, N '50, 2
———, engineering schools' contribution to research, Ja '51, 2
———, future of research, S '50, 2
———, industrial and governmental research, My '50, 2
———, southern industrialization, trained manpower from Georgia Tech, Mr '51, 2
Varner, John Edward,
———, a critical study of the copper spark method of spectrographic analysis, Ja '51, 16
Varnishes, N '50, 4
———, a study of physical properties, S '50, 14
V. D. Anderson solvent extraction unit, see Anderson
Vegetable oils, extraction, S '50, 12
Velocity modulation, S '50, 18
Very low temperature research, S '50, 11
Vision and hearing tests, importance in employee selection, S '50, 20
Vitrification, Mr '51, 13

W

Wage incentive payment for multiple machine assignments, My '50, 11
Waste, radioactive, disposal, N '50, 14
Water and sewage analysis, S '50, 14
Water, channel,
———, used in tests of airfoils, S '50, 21
———, used in tests of wind tunnel shapes, S '50, 22
Weil, B. H.,
———, the Georgia Tech Library as a tool for science and industry, N '50, 5
Windows, testing, S '50, 15
Wind tunnel,
———, design, supersonic, problems, S '50, 22
———, studies, S '50, 16
Wings, swept-back, maximum lift coefficient, S '50, 16
Winthron, Stanley Oscar,
———, a study of the condensation of thioisophthalaldehyde, Ja '51, 17
Wood distillation, S '50, 14
Woodward, L. A.,
———, electron microscopy, Mr '51, 3

Y

Yarns, physical study, S '50, 14

EXPOSURE TESTING

Continued from Page 4

obtained under different sets of conditions, and the effects of these differences cannot be evaluated quantitatively with the data at hand. In most cases close liaison is necessary to establish good correlations between the work of different investigators.

Accelerated Tests

To complete this discussion mention should be made of accelerated testing techniques. For the most part, accelerated tests have not been found very reliable in predicting paint film performance under normal conditions.[13 14 15 16] As a rule, all of the physical and chemical changes which produce paint film failures are not accelerated at the same rate by these tests. Thus a film might be completely eroded in an ac-

celerated test before such failures as cracking or peeling can be observed. This limitation is characteristic of mechanical (carbon arc and water spray) weathering machines, and is also present to a lesser extent in other accelerated techniques such as 45° exterior exposures for house paints and exposures in south Florida.

It is claimed that south Florida exposures which usually enhance cracking and peeling can be well correlated with exposures in other locations. To do so requires use of an experimentally determined compensation factor for each type of film failure. These factors can be determined only by comparison of Florida exposures with normal exposures in the locality considered.

There is still a great need for a reliable accelerated weathering technique that could be used to predict film performance under any desired climatic conditions, and some work has already been done along this line. As exterior paints continue to improve in durability, the need for accelerated weathering tests becomes increasingly greater to permit evaluation within a reasonable time. It therefore seems inevitable that a reliable technique will be developed in the future.

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QUANTITATIVE RESEACH

Continued from Page 2

Speaking of his food preservation work at Georgia Tech, Kethley says that it "has been based on the theory that fundamental studies on processes and causes will yield information, which can be applied to practical considerations, and that such a long-range program will produce more practical and useful results in a shorter length of time than will short-term, so-called applied research." This statement admirably sums up the function of quantitative research.

Today there is a greater need than ever for quantitative facts, the basic building blocks to provide the foundations of a superior technology for defense and of an expanding economy in peace. Industry must concentrate upon fairly short-range applied research with the promise of quick results. But applied research draws upon long-range studies for fundamental data. Thus, the need for nonprofit institutions to perform quantitative research is as obvious as is the necessity for such research, itself. Fortunately, as a part of its unparalleled educational system, our nation possesses the type of quantitative research personnel and facilities required. Basic studies prosecuted in universities and their allied experiment stations combined with the applied research and productivity of American industry are our key to a secure and ever brighter future.